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THE KEEPING QUALITIES OF CONCENTRATED DISTILLERS' SOLUBLES WHEN USED AS A REPLACEMENT OR EXTENDER FOR MOLASSES IN MIXED FEEDS.

by

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Early in 1945 developments in the feed-supply situation indicated that there was a need for a more diversified outlet for the marketing of distillers' solubles produced from grain mashes used in the manufacture of alcohol and at the same time for an extender to augment the inadequate supplies of molasses needed in mixed feeds. An investigation was accordingly undertaken by means of small scale laboratory tests of the practicability of using concentrated or semisolid distillers' solubles alone or in combination with molasses as an ingredient in feed mixtures. Among the questions to be answered was that of keeping qualities, or tendency to mold. The known high moisture content of semisolid distillers' solubles naturally suggested that the resulting increase in moisture content of the feed mixture might be a serious obstacle. Reduction of the moisture content of the solubles to equal that of molasses, however, was seemingly out of the question because of limitations of the evaporators and the thick viscous nature of the product.

It has been observed that dried distillers' solubles are often highly hygroscopic. Such behavior if manifested in a mixture of condensed solubles and other feeds might increase mold growth to a considerable extent. Finally, one of the attributes of molasses as an ingredient in feed mixtures is its ability to reduce the dustiness. To be of full value as a replacement for molasses, therefore, the solubles should possess comparable properties.

In view of these circumstances there was a need for information on the limits of moisture content, temperature, relative humidity, and time in their relation to mold growth and spoilage of the feed. Conversely, the question of inhibiting effects of mixing solubles with blackstrap or corn sugar molasses on the spoilage of feed was considered. A secondary problem in the handling of the corn sugar molasses, namely the tendency to crystallize and solidify, was also considered as possible of alleviation through admixture with the condensed solubles.

The investigation was, therefore, designed to study the effect of these factors just named, especially the relative humidity and temperature of the storage environment, on the rate of spoilage of feed mixtures containing various amounts and combinations of molasses and solubles products.

Procedure

Two different samples of concentrated distillers' solubles were used in the experiments. The first one was a semisolid product containing about 35 percent solids concentrated according to the conventional method from a grain mash stillage. A chemical analysis of this sample showed the following:

Moisture	64.42	percent
Crude protein	10.58	"
Ash	2.23	"
Fat	2.05	"
Crude fiber	1.83	"
Nitrogen-free extract	18.89	"

The second one was a semisolid distillers' solubles prepared by a special process from a grain mash stillage. It differed from the first product in having more of a syruplike consistency due apparently to greater fineness of the particles. Analytical data on this sample were:

Moisture	50.49	percent
Crude protein	13.36	"
Ash	5.26	"
Fat	0.36	"

The blackstrap molasses had a total solids content of 75.5 percent while the corn sugar molasses contained 76.4 percent. The feed mixture was made up of 25 parts each by weight of ground corn, ground oats, and wheat bran, and 10 parts of soybean meal.

The first part of the study consisted of a series of experiments dealing with the influence of humidity on mold growth. The general plan of the experiments was to mix varying percentages of solubles, molasses, or their combinations into the feed mixture and determine the length of time required for mold growth to be observable under the different conditions of storage. In the usual case pint jars were filled with the mixture to be studied and placed in desiccators provided with appropriate solutions to maintain the desired humidity. To the basal feed mixture was added either 5 or 10 percent of the first sample of semisolid solubles, of blackstrap molasses, of corn molasses or equal parts of any two of these products. In other cases these feed mixtures were either stored in paper bags under prevailing laboratory humidity and temperature conditions or were spread out in thin layers in Petri dishes placed in the desiccators.

Although the initial moisture contents of the feed mixtures in the foregoing varied through a considerable range and thereby had an influence on the results obtained, the second phase of the study was devoted to the question of moisture content. The additions of solubles (first sample) and molasses were made at levels to produce mixtures with moisture contents in what was considered the critical range of 10 to 17 percent. The samples were placed in fruit jars which were sealed and placed in temperature-controlled rooms.

Because so many of the samples molded in a comparatively short time under the conditions outlined, further experiments were conducted on the keeping qualities of solubles and molasses in different combinations ranging from all solubles to all molasses. A limited amount of the second sample of solubles had become available when this phase of the work was conducted. Accordingly it was used in tests on keeping quality which included fermentation as well as mold formation. In the fermentation tests, the mixtures of solubles and molasses were placed in fermentation tubes incubated at 68° and 98° F. The samples for mold growth were placed in Petri dishes.

Relative humidity was maintained at 66 percent, 74 percent, 80 percent and 100 percent in sealed desiccators in the following manner: 100 percent humidity by using water in the desiccators, 66 and 74 percent by the use of a saturated solution of KNO_2 at 98° F. respectively and 80 percent by the use of a saturated solution of $(NH_4)_2SO_4$. The humidity as recorded in the tables was determined by the use of wet and dry bulb thermometers.

Temperatures were maintained at 68° F. and 98° F. in constant temperature rooms. The temperatures did not vary by more than plus or minus two degrees during the course of the experiments.

Results

The relation of relative humidity to mold growth.

In the initial study, a basal feed mixture was used which contained 13.2 percent moisture due in large part to the high moisture content of the corn. The semisolid solubles (35 percent solids) and the two types of molasses were incorporated by hand into the basal mixture in the combinations and at the levels shown in tables 1 and 2. Sets of samples in pint jars were stored at 100 percent relative humidity and at temperatures of 68° and 98° F. while other sets were held at 74 percent relative humidity and 68° F. and at 66 percent relative humidity and 98° F. When any sample showed mold growth, it was removed, the entire contents of the jar were mixed and portions taken for moisture determination. Samples remaining after 42 days were treated in the same manner.

Data on initial and final moisture content and the time for mold growth to appear are shown in tables 1 and 2. In the case of the samples held at 100 percent relative humidity (table 1), spoilage was very rapid. It is of interest to note that all samples picked up additional moisture even though the initial content was as much as 19.6 percent. Furthermore temperature was not an important factor in the uptake of moisture. At lower humidity levels a different order of results was obtained as shown in table 2. Only 3 of the 22 samples showed mold growth after 42 days. All samples lost moisture, those stored at 98° F. drying out more than those stored at 68° F. The samples which spoiled had an initial moisture content of 17.2 percent or over. All three were from the solubles groups.

Three-pound lots of some of the same samples listed in tables 1 and 2 were placed in paper bags and left at ordinary room temperature and humidity for 15 weeks (April-July). The samples represented were those containing 10 percent of added solubles or molasses or their mixtures. No mold was observed in any case and all 5 samples came to an equilibrium in moisture as indicated by a final content of approximately 8.5 percent.

In view of the rapidity with which spoilage occurred in the samples with a high initial moisture content and stored under conditions of high humidity, a new batch of basal feed mixture was prepared which was dried down to a moisture content of 1.11 percent. Molasses and solubles were added at the same levels and combinations which were used in the tests just described. All the samples were stored at 100 percent relative humidity half at 98° F. and half at 68° F. At the first signs of mold, the top 1.5 inches of material were removed from each jar and analyzed for moisture. The data on mold growth and change in moisture content are shown in table 3. It will be noted that mold growth was observed in 7 to 13 days in the samples held at 98° F. and in 9 to 17 days in those held at 68° F. The difference between solubles and molasses is not striking although the solubles tended to accelerate spoilage presumably because this material supplied more moisture at the start of the test. It is interesting to observe the pick-up in moisture content in all samples and the relatively small range in values, namely 12.4 to 15.6 percent.

Attempt was next made to select a relative humidity intermediate to the extremes already used which would not cause any pronounced shift in moisture content of the samples either hydration or dehydration. Accordingly samples

of intermediate moisture content were stored at temperatures of 68° F. and 98° F. in an atmosphere maintained at 80 percent relative humidity. At the first sign of mold, the top one-half inch of material was analyzed for moisture. The results are shown in table 4. While the samples gained moisture and molded, the rate of spoilage was greatly reduced over that in samples stored at 100 percent relative humidity. A temperature of 68° F. generally favored retardation of mold growth. The addition of distillers' solubles appeared to be about as safe as the addition of molasses. The use of equal parts of solubles and either blackstrap or corn molasses seemingly caused more rapid mold formation than the use of any one product alone.

A further test of conditions of 80 percent relative humidity and 68° F. and 98° F. was carried out on samples placed in Petri dishes in thin layers. The data on observation of mold growth were recorded and at the end of 6 weeks, after the samples had come to moisture equilibrium, they were analyzed for moisture content. As indicated in table 5, all samples held at 68° F. molded in 18 to 20 days while those held at 98° F. molded in 11 days.

Moisture content and mold development

In most of the tests thus far described, the humidity of the surrounding atmosphere was controlled at an arbitrary level. Under practical conditions where large masses of mixed feeds are involved only a small portion constituting the surface layer may be exposed to the atmospheric moisture. Accordingly a test was carried out on samples of feed mixtures containing solubles and molasses at different levels and combinations which were stored in sealed jars at temperatures of 68° and 98° F. As indicated in table 6, a sufficient number of samples was provided to give a series in which the moisture content did not differ in most cases by more than approximately 1 percent. It will be observed that mold growth was slow in developing in those samples stored at a temperature of 68° F. when the moisture content did not exceed 14 percent. There was evidence that spoilage occurred earlier in those samples containing distillers' solubles than in those containing molasses or mixtures of solubles and molasses when moisture content was held constant. The same was true in the case of the samples stored at 98° F. where 24 days was the time limit before mold was observed in a sample containing 13.9 percent moisture and made up with 10 percent of distillers' solubles as compared to 41 days for a sample containing 17 percent of a mixture of distillers' solubles and blackstrap molasses but with the same moisture content and 95 days for a sample containing 29.5 percent of blackstrap molasses and 14 percent moisture.

Fermentation and mold growth of solubles and molasses.

The apparent advantage in keeping qualities of the samples containing molasses prompted the further testing of a series of combinations of solubles and blackstrap or corn-sugar molasses. The ratios of solubles to blackstrap were (a) 7: 3, (b) 5: 5, (c) 3 : 7 and (d) 1 : 9 respectively. Each mixture was inoculated with mold spores and yeast cells. The mixtures were stored in half-pint jars sealed except as otherwise noted, two jars were used for each mixture and storage condition, one jar was filled and the other half filled. The storage temperatures used were 68° and 98° F. Control samples of solubles, blackstrap molasses and corn sugar molasses were also stored under the same conditions. The mixtures of blackstrap molasses and solubles stored in sealed jars fermented quite rapidly, samples containing 50 percent or more of solubles fermented within 10 days at both temperatures and even the samples

containing only 1 part of solubles fermented within 37 days regardless of temperature. Some of the samples also molded within a few days. Samples containing equal parts of blackstrap molasses and solubles which were kept in unsealed containers formed a tight scum over the surface which seemed to inhibit mold growth and fermentation. No mold growth and only very slight fermentation if any developed in the solubles - corn sugar molasses mixtures during a storage period of 85 days. Mixtures of solubles and corn sugar molasses containing more than 30 percent solubles generally separated into two layers on long standing, a liquid layer forming at the bottom and a semisolid layer on top. No difference was noted between the jars that were full or half full. The blackstrap and corn sugar molasses control samples did not ferment during a storage period of 50 days, but the sample of solubles deteriorated rapidly molding within three to six days and fermenting within 10 days. It was found that corn sugar molasses crystallized on standing, time seeming to be the only factor involved.

In view of the results obtained with the first sample of solubles, it was decided to test also the second sample in a short series of fermentation and molding tests without admixture with the grain feed. It will be recalled that the second sample contained 50 percent of solids and therefore possessed some advantage over the first one in this respect. Accordingly a series of fermentation tubes were set up at 98° and 68° F., containing 100 percent solubles, 50 percent solubles plus 50 percent blackstrap molasses and 50 percent solubles plus 50 percent corn sugar molasses. Petri dishes containing the above mixtures and inoculated with mold spores were also stored at the same temperatures. Some fermentation was observed in the mixture containing 50 percent solubles and 50 percent blackstrap molasses stored at 98° F., and the sample containing solubles alone kept at 98° F. fermented slightly. No fermentation was observed in the mixture of 50 percent solubles and 50 percent corn sugar molasses at 98° F., and no fermentation was observed in any of the mixtures at 68° F., during a storage period of 65 days. No molding was detected on any of the samples during the same period.

Discussion

From this study it is evident that the relative humidity under which the feed mixtures are stored is a very important factor in their deterioration through mold growth. There was no significant mold growth during a six week period at humidities up to 76 percent except in the samples containing over 17 percent of moisture, but mold growth did occur in all samples at a humidity of 80 percent or above even though the moisture content was as low as 13.5 percent at the end of the experiment. Snow, Crichton, and Wright (1) and Galloway (2) have also found that the relative humidity rather than the moisture content of the substratum, is the important factor in the rate of mold germination. Galloway found that molds of the Aspergillus glaucus, A. candidus and A. versicolor series could develop at 75-80 percent relative humidity. Snow, et al, found that mold growth took place relatively quickly on all feedingstuffs stored at 75-100 percent relative humidity and that below 75 percent humidity mold growth only developed after a very prolonged latent period.

In the present experiments, high temperatures accelerated the rate of mold growth, thus confirming the work of Snow, et al. The safe limit of moisture content thus depends on the composition of the feed mixture and on the storage conditions. When stored in sealed jars at a temperature of 98° F.

mold developed fairly rapidly above a moisture content of 13 percent in samples containing solubles, while mixtures containing both solubles and molasses molded at a slower rate and at a higher moisture content, and mixtures containing 36 percent or more of molasses did not mold even though they contained up to 17 percent moisture. It would seem that about 12 percent moisture is the top limit for mixtures containing solubles, unless the feed can be stored under conditions favorable for the loss of moisture. At a storage temperature of 68° F., there is a retardation in the rate of molding and consequently a higher moisture content of approximately 13 percent for a mixture containing solubles alone (Sample 1) and 15 percent for a mixture containing equal parts of solubles and molasses can be tolerated. Because of the difference in moisture content, it follows that solubles sample 2 should be safer to use than sample 1.

The moisture content at equilibrium is greatest in the mixtures containing molasses. The average moisture content of four samples containing solubles after reaching equilibrium at 80 percent relative humidity and 68° F. was 16.2 percent while under the same conditions the average moisture content of two samples containing both solubles and molasses was 18.0 percent. Snow, et al, found that the level and shape of the water absorption curves were closely related to the amounts of soluble carbohydrates and protein present and that fiber exerted a depressing effect on the water uptake.

Solubles appear to be less effective in reducing the dustiness of feed than molasses. The addition of 10 percent of condensed solubles produced a feed that was less dusty than the basal feed but was dustier than a mixture containing 5 percent molasses. A mixture containing 5 percent solubles and 5 percent molasses was less dusty than the 5 percent molasses but dustier than the 10 percent molasses sample.

Under the conditions employed, the keeping qualities of feeds containing mixtures of solubles and blackstrap or corn sugar molasses were superior to those containing solubles alone. However the rapid deterioration of solubles (Sample 1) and molasses mixtures indicates the necessity of using them in feed mixtures within a short time after preparation.

The physical characteristics of condensed solubles such as represented in the first sample present problems of shipment, storage and mixing. It was too thick and viscous to handle readily if allowed to cool. Consequently molasses or feed mixtures would have to be transported to the distillery and the hot solubles from the evaporators would have to be run in while it was still liquid.

The second solubles sample with a moisture content of approximately 50 percent yet possessing free flowing properties similar to molasses appears to offer much better possibilities for mixing with feeds. It not only remains relatively free of mold when stored alone but has the advantage of adding less moisture when incorporated in a feed mixture and consequently reduces the likelihood of molding as compared to the other sample.

Summary

(1) It appears that condensed distillers' solubles can be used as a substitute for a part at least of the molasses used in feed mixtures without a

serious spoilage hazard providing the moisture content is kept below 13 percent and high storage temperatures are avoided.

(2) The use of ordinary concentrated distillers' solubles of 35 percent solids content appears to be chiefly limited by its high moisture content.

(3) The physical characteristics of solubles of this type present additional problems in shipment, storage and mixing.

(4) The distillers' solubles were also less effective in reducing the dustiness of feed mixtures than molasses.

(5) The keeping qualities of feeds containing mixtures of blackstrap or corn sugar molasses were superior to those containing solubles alone. Nevertheless the rapid deterioration of solubles (Sample 1) and molasses mixtures necessitate their use in feed mixtures within a short time after preparation.

(6) Distillers' soluble with a total solids content of about 50 percent appears to offer much better possibilities for mixing with feeds than the grain distiller's solubles containing about 35 percent solids, due to its free flowing properties, lower moisture content and better keeping qualities.

References

(1) D. Snow, M. H G. Crichton and N. C. Wright, Ann. Applied Biology, V. 31, 102-10, 111-16 (1944).

(2) L. D. Galloway, J. Textile Inst. V. 26, Tl 23-9 (1935).

Table 1. Effect of temperature on formation of mold in feed mixtures of moderately high moisture content stored at a relative humidity of 100 percent.

Ingredient	Percent added to basal mixture	Initial moisture content	Temperature of 68°F.		Temperature of 98°F.	
			Final moisture content	Time when mold observed	Final moisture content	Time when mold observed
		Percent	Percent	Days	Percent	Days
Distillers' solubles	5	15.0	17.5	5	17.3	4
	10	17.2	19.3	5	19.0	3
	15	19.6	21.8	5	21.8	5
Blackstrap molasses	5	13.5	15.8	8	16.8	5
	10	14.2	17.0	7	18.0	4
Corn molasses	5	13.4	16.6	7	16.1	6
	10	13.9	16.9	6	16.9	6
Distillers' solubles and blackstrap molasses 1/	5	14.4	16.9	7	16.8	4
	10	15.7	18.6	7	19.3	3
Distillers' solubles and corn molasses 1/	5	14.5	16.9	6	16.8	6
	10	15.7	18.0	6	17.9	6

1/ Equal parts

Table 2. Effect of relative humidity and temperature on change in moisture and formation of mold during storage of feed mixtures.

Ingredient	Percent added to basal mixture	Initial moisture content	Relative humidity of 74 percent and temperature of 68°F.		Relative humidity of 66 percent and temperature of 98°F.	
			Final moisture content	Time when mold observed	Final moisture content	Time when mold observed
		Percent	Percent	Days	Percent	Days
Distillers' solubles	5	15.0	13.1		9.7	
	10	17.2	15.0	42*	10.2	
	15	19.6	17.2	42	11.4	42
Blackstrap molasses	5	13.5	12.2		10.0	
	10	14.2	12.5		10.1	
Corn molasses	5	13.4	12.0		10.2	
	10	13.9	12.3		9.8	
Distillers' solubles and blackstrap molasses 1/	5	14.4	12.4		10.5	
	10	15.7	13.5		10.6	
Distillers' solubles and corn molasses 1/	5	14.5	12.9		10.7	
	10	15.7	13.9		11.1	

1/ Equal parts

* Mold was found in center of jars at end of experiment.

Table 3. Effect of temperature on change in moisture and formation of mold in feed mixtures with a low moisture content stored at a relative humidity of 100 percent.

Ingredient	Percent added to basal mixture	Initial moisture content	Temperature of 68°F.		Temperature of 98°F.	
			Moisture content when mold observed	Time when mold observed	Moisture content when mold observed	Time when mold observed
		Percent	Percent	Days	Percent	Days
Distillers' solubles	5	4.9	12.8	11	13.2	8
	10	8.1	15.6	11	14.1	8
	15	9.2	15.2	11	14.7	7
Blackstrap molasses	5	2.9	12.7	17	13.0	8
	10	4.2	13.3	16	14.8	9
Corn molasses	5	3.5	15.6	13	14.8	13
	10	4.6	13.8	16	15.6	9
Distillers' solubles and blackstrap molasses 1/	5	3.9	12.4	10	14.2	13
	10	6.3	15.5	9	14.3	10
Distillers' solubles and corn molasses 1/	5	3.6	14.3	14	14.2	7
	10	5.9	15.0	13	14.6	7

1/ Equal parts

Table 4. Effect of temperature on change in moisture and formation of mold in feed mixtures with low moisture content and stored at a relative humidity of 80 percent.

Ingredient	Percent added to basal mixture	Initial moisture content	Temperature of 68°F.		Temperature of 98°F.	
			Moisture content when mold observed	Time when mold observed	Moisture content when mold observed	Time when mold observed
		Percent	Percent	Days	Percent	Days
Distillers' solubles	5	7.7	15.0	39	13.6	31
	10	10.1	14.9	38	13.6	29
	15	13.2	14.9	33	13.8	22
Blackstrap molasses	5	12.2	15.2	33		
		10.0			13.4	29
	10	12.5	15.3	33		
		10.1			14.0	29
Corn molasses	5	12.0	15.5	36	13.8	29
		10.2				
	10	12.3	15.6	37	14.3	29
		9.8				
Distillers' solubles and blackstrap molasses [1]	5	12.4	15.0	21		
	10	10.5			13.5	21
		13.5	15.4	10		
		10.6			13.9	21
Distillers' solubles and corn molasses [1]	5	12.9	15.0	29		
	10	10.7			14.0	19
		13.9	15.6	10		
		11.1			13.5	12

1/ Equal parts

Table 5. Effect of temperature on change in moisture and formation of mold in feed mixtures stored in thin layers at a relative humidity of 80 percent.

Ingredient	Percent added to basal mixture	Initial moisture content	Temperature of 68°F.		Temperature of 98°F.	
			Final moisture content	Time when mold observed	Final moisture content	Time when mold observed
		Percent	Percent	Days	Percent	Days
Distillers' solubles	8	12.9	16.1	19	14.0	11
	10	13.9	16.2	18	14.0	11
	12	14.9	16.3	18	13.8	11
	14	16.0	16.3	18	14.1	11
Distillers' solubles and blackstrap molasses 1/	23	15.9	17.2	20	14.6	11
	26	16.9	18.7	19	15.0	11

1/ Equal parts

Table 6. Effect of temperature on formation of mold in feed mixtures stored in sealed containers.

Ingredient	Percent added to basal mixture	Initial moisture content	Temperature of 68°F. Time when mold observed	Temperature of 98°F. Time when mold observed
		Percent	Days	Days
Distillers solubles	5	10.6	*	*
	8	12.9	70	35
	10	13.9	41	24
	12	14.9	20	13
	14	16.0	18	13
	16	17.0	18	9
Blackstrap molasses	29.5	14.0	†	95
	36	15.0	†	†
	43	16.0	†	†
	50	17.0	†	†
Corn molasses	10	9.8	*	*
Distillers' solubles and blackstrap molasses 1/	8	10.8	**	**
	10	11.7	*	*
	11	11.9	**	**
	14	13.1	**	100
	17	13.9	100	41
	20	15.0	62	35
	23	15.9	59	42
	26	16.9	45	31
Distillers' solubles and corn molasses 1/	10	11.9	*	*

1/ Equal parts
* No mold in 42 days.
† No mold in 95 days.
** No mold in 109 days.

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